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(54) MEMBER WITH SOLID LUBRICANT FILM

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a member with solid lubricant film, causing no deterioration in adhesion between solid lubricant film and member even if an inexpensive solid lubricant film is used, capable of giving low coefficient of friction over a long period, and also capable of keeping high wear resistance.

SOLUTION: A primary layer composed of hard film having a large number of micropores of 0.1-3 μm inside diameter and a secondary layer composed of solid lubricant film are formed. The surface roughness Rmax of the primary layer before the formation of the secondary layer is regulated to ≤1 μm. As the solid lubricant film of the secondary layer, a film of MoS2, WS2, NbS2, mica, Sb2O3, BN, WSe, MoSe2, Au, Ag or the like is formed by the cathode arc ion plating method, the sputtering method or the shot peening method.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention] This invention relates to the member with solid-state lubricating film which improved abrasion resistance in more detail with low coefficient of friction useful as machine part applications, such as sliding wear components and a tool, about the member with solid-state lubricating film excellent in lubricity and abrasion resistance.
[0002]
[Description of the Prior Art] As a means which reduces coefficient of friction of a tool or a machine part, and raises an antiwear characteristic, the approach of forming solid-state lubricating film in a member front face is learned. For example, apply an oil to a machine bill-of-materials side, it covers to it in the film and oil which distributed the lubrication particle, or **, such as MoS2 film, are formed in it by the sputtering method.
[0003] However, by the approach of these former, solid-state lubricating film did not form a member and a diffusion layer, but the adhesion force of a member and solid-state lubricating film was weak. Moreover, since the film itself did not have abrasion resistance, when long duration use was carried out, the film was worn out, and there was a fault of being exfoliated, worn out and lost from a member front face.
[0004] Moreover, since solid-state lubricating film, such as MoS2, generally had the weak adhesion force and hygroscopicity was high, the film itself adsorbs moisture, it has viscosity and tended to exfoliate immediately from a member.
[0005] Furthermore, in the shaft and bearing of a pump which carry out high-speed rotation, especially high abrasion resistance and high lubricity (low coefficient of friction) are required, and what is further used underwater and in hot water needs the higher adhesion force of solid-state lubricating film. For this reason, the property of the above-mentioned conventional solid-state lubricating film is inadequate, and ceramic material expensive until now, thermal-spraying material, etc. had to be used.
[0006]
[Problem(s) to be Solved by the Invention] Then, even if it uses cheap solid-state lubricating film, the adhesion force of solid-state lubricating film and a member does not decline, but low coefficient of friction is obtained over long duration, and this invention aims at offering the member with solid-state lubricating film which can moreover hold high abrasion resistance.
[0007]
[Means for Solving the Problem] The member with solid-state lubricating film of this invention for solving the above-mentioned purpose is characterized by forming the 2nd layer, the 1st layer and solid-state lubricating film of the hard film which has many detailed holes with a bore of 0.1-3 micrometers.
[0008] It is desirable for surface roughness Rmax of the 1st layer before forming the 2nd layer in the member with solid-state lubricating film of this invention to be 1 micrometer or less.
[0009] As for the hard film of the 1st layer, it is desirable that they are the nitride film by the cathode arc ion plating method, the carbon nitride film, or the carbide film. It is good to set bias voltage to -500--

1500V by the cathode arc ion plating method.
[0010] As for the solid-state lubricating film of the 2nd layer, it is desirable that they are MoS2 film formed by the ion plating method, the sputtering method, or the shot-peening method, WS2 film, NbS2 film, the mica film, 2OSb3 film, BN film, the WSe film, MoSe2 film, Au film, or Ag film. It is good to set bias voltage to -500--1500V by the ion plating method, the sputtering method, or the shot-peening method.
[0011]
[Embodiment of the Invention] What is used for machine part applications, such as sliding wear components and a tool, from the former is applicable to the member of the member with solid-state lubricating film of this invention from the former. For example, they are a cutting tool, the SKD steel for metal mold, SKH steel, SKS steel, SC steel of a machine structural steel worker, etc.
[0012] Although the hard film which has many detailed holes with a bore of 0.1-3 micrometers as the 1st layer is formed in a member front face, this is formed for the purpose of raising the adhesion of the hard film and solid-state lubricating film.
[0013] If many 0.1-3-micrometer detailed holes (depression) are formed in a hard film front face, solid-state lubricating film will adhere by using the depression as a nucleus, and the adhesion force will improve according to an anchor effect. Moreover, even if it wears solid-state lubricating film out, when partner material is contacted, and the solid-state lubricating film deposited on a depression is dug up and oozes out, the solid-state lubricity on the front face of a member also becomes maintained for a long time.
[0014] It is because solid-state lubricating film enters into a hole and is not easily removed from a front face because setting the bore of a detailed hole to 0.1-3 micrometers considers as the bore of this range. About 0.001% of abundance of thickness is [this detailed hole] moreover, desirable at 10-20 micrometers 0.01% per area in 5 micrometers.
[0015] The hard film consists of a nitride, carbon nitride, or carbide desirably, CrN, TiN, TiCN, TiAlN, etc. can specifically be used, it is considering as such the quality of the material, and abrasion resistance and low coefficient of friction are obtained. The hard film can be formed for example, by the cathode arc type ion plating method.
[0016] This cathode arc type ion plating method uses as cathode the metal target which is an evaporation source, by making a chamber into an anode plate, among these, makes a member front face carry out the laminating of the compound which caused arc discharge, was made to ionize metallic fumes, ionized reactant gas by the collision with ion by one side, and was generated by the metal ion and reaction gas ion, and forms the film. For example, what is necessary is just to introduce the reactant gas containing a nitrogen atom in a chamber, when forming a nitride.
[0017] The metaled ionization rate of the cathode arc type ion plating method is high, and since it can install two or more evaporation sources in the four directions in a chamber, it could form the film of uniform thickness in the member of bipolar membrane, a large-scale configuration, or a complicated configuration, and is fit for hard film formation of these members. Before film formation, if bias voltage applied between a target and a member is set to -500--1500V, a metal will adhere to a member and will serve as irregularity of the 5 micrometers of the maximum granularity. If hard film, such as a nitride, is besides formed, the hole of a large number with a bore of 0.1-3 micrometers can be formed.
[0018] If bias voltage is made low with less than [-500V], the number of holes will decrease and the effectiveness of this invention will decrease. On the other hand, although the number of holes will increase if bias voltage is made high exceeding -1500V, the surface roughness of a member also increases and the dimensional accuracy as a product falls.
[0019] Solid-state lubricating film is formed on the 1st layer at the 2nd layer. MoS2, WS2 and NbS2 of a sulfide, the mica of an oxide, Sb 2O3, BN of a nitride, WSe of a selenide, MoSe2, metaled Au, Ag, etc. are mentioned to the ingredient of solid-state lubricating film. What is necessary is just to form this solid-state lubricating film by for example, the ion plating method, the sputtering method, the shot-peening method, etc.
[0020] Surface roughness Rmax of the 1st layer before forming the 2nd layer may be 1 micrometer or

less desirably by surface polish processing etc. It is because can remove the molten metal formed in the hard film front face called "drop let" by this, it can decrease, the touch area of the hard film and solid-state lubricating film increases and the adhesion force improves. Moreover, drop let's exfoliating from the film and shaving solid-state lubricating film can also be prevented. As surface polish processing, diamond paper polish, a surface-grinding machine, buffing, etc. can be used.

[0021] Solid-state lubricating film enters the interior of the hole formed in the hard film, and combines mechanically the adhesion force of a member and solid-state lubricating film according to an anchor effect. This bonding strength becomes stronger than the conventional approach.

[0022] A formation case can use direct-current (DC) sputtering and the (RF RF) sputtering method for solid-state lubricating film further by the sputtering method. Although inert gas, such as Ne, Ar, and Kr, can be used for discharge gas, Ar gas with easy handling is desirable. For example, if discharge gas pressure is set as 1-4Pa, it will be precise and solid-state lubricating film with the high adhesion force will be formed. What is necessary is just to consider discharge power to a target as per [100-500W] target.

[0023] As for the thickness of solid-state lubricating film, it is desirable to be referred to as 0.1-4 micrometers. It is because membranous shearing becomes easy to happen, many Ar molecules are contained in the film, if lubricity sufficient in less than 0.1 micrometers is not obtained but exceeds 4 micrometers, so lubricating properties also deteriorate.

[0024] What is necessary is just to inject the mixture which mixed solid lubricant powder and a synthetic-resin particle at a fixed rate on a member front face with a dry type blasting machine, when forming solid-state lubricating film by the shot-peening method. Solid lubricant powder collides and adheres to a member front face, and a resin particle is repelled at the same time it collides with a front face, and it flies away with a gas style. At this time, a resin particle drives in and rubs in the solid lubrication fine particles adhering to a front face, and the good solid-state lubricating film of adhesion with a front face is formed. In addition, since it is hard to form the thick film of solid-state lubricating film in the shot-peening method, 1 micrometer or less in thickness is suitable.

[0025]

[Example] Example 1 ... Tool steel (SKH51) of 2mm in thickness and 20mm angle was used as the member, and the film of the 1st layer was formed using the cathode arc type ion plating system by the multi-arc company. Cr metal was used for the target. After cleaning a member ultrasonically in ethanol, it set in the vacuum chamber and the inside of a chamber was exhausted to 2x10 to 5 or less Torrs. The bias voltage of -1000V was impressed between the target and the member, the member front face was washed and heated by metal bombardment of Cr metal, Cr ion was driven in, and member temperature was made into 450 degrees C.

[0026] Next, N2 gas was introduced to 50mTorr(s) in the chamber, 10 micrometers of CrN film were formed by bias voltage-300V, and it could be the 1st layer.

[0027] Observation of the front face of the formed CrN film formed 100-200 1mm per two and holes with a bore of 0.2-3 micrometers.

[0028] Then, the lubricant film of the 2nd layer was formed using RF magnetron sputtering system (the product made from Japanese ** Anelva, Inc., SPF530H). A disk mold molybdenum disulfide (MoS2) target with a diameter of 5 inches is set to the predetermined location in equipment, and the degree of vacuum in a chamber is 5x10-4. Vacuum suction was carried out until it was set to Pa, and Ar gas was introduced to 0.4Pa. The 300W (2.37 W/cm2) injection of the discharge power was carried out, and it formed 1.2 micrometers of MoS(s)2 in Ar gas ambient atmosphere, and made them the 2nd layer.

[0029] The ball-on disk trial with a diameter of 12mm which made the obtained member with solid-state lubricating film 10 Ns of loads and peripheral-velocity 0.5 m/sec using the ball with a diameter of 6mm made from SUS440C estimated the count whose MoS2 is lost as a count of durable friction. The count of durable friction was 450x102 times, and coefficient of friction was 0.1. Moreover, Vickers hardness was 1800HV. Moreover, the cutter knife was used for the member front face for the adhesion force, the blemish with a spacing [of 1mm] and a die length of about 10mm was formed in the shape of a grid, adhesive tape was stuck on the part, and no exfoliation was seen, when this was removed at a stretch and

exfoliation of the film was observed.
[0030] The conventional example 1 ... The 1st layer of an example 1 and this thickness was formed without using an electron beam type ion plating system for the same member as an example 1, and forming a hole, and the 2nd layer of MoS2 was formed like the example 1. When it was similarly estimated as the example 1, the count of durable friction was 270x102 times, and coefficient of friction was 0.15. Moreover, Vickers hardness was 900HV. About 50% of the film exfoliated in the same adhesion force evaluation as an example 1.
[0031] Example 2 ... The film of the 1st layer was formed like the example 1 except having used stainless steel (SUS304) of 2mm in thickness, and 20mm angle as the member, and having set the CrN film to 20 micrometers. Observation of the front face of the formed CrN film formed 100-200 1mm per two and holes with a bore of 0.2-2 micrometers.
[0032] Buffing of this member front face was carried out, and surface roughness Rmax was set to 0.08 micrometers. Distance of an injection air gun and a member was made this ground member for mixed powder (740g of MoS2 powder, and 50-micrometer resin bead 320g), 100mm and injection Ayr ** were made into 3 kgf/cm2, it sprayed and the 2nd layer membrane which consists of MoS2 film by the shot-peening method was formed. The thickness of the 2nd layer was 1 micrometer.
[0033] No membranous isolation was seen in the same adhesion force evaluation as an example 1.
[0034] Moreover, in the Falex trial of load 200kgf and rotational-speed 50rpm, even if 1000 seconds had passed since rotation initiation, torque value hardly changed and solid-state lubricating film did not exfoliate, either.
[0035] When the Falex trial estimated the member of the conventional example 1 similarly, after [rotation initiation] MoS2 powder scattered and torque value increased gradually after 800-second progress.
[0036] Example 3 ... 10 micrometers of CrN film were formed as the 1st layer like the example 1, and buffing removed and estimated the drop let generated on the front face. Surface roughness Ra in front of buffing was 0.08 micrometers, and after buffing was 0.03 micrometers.
[0037] The solid-state lubricating film which moreover consists of Ag film with the Shinko Seiki pierced earring type ion plating system was formed. Ag ingot of a trapezoid mold was put into the crucible made from copper-molybdenum, membranes were formed for 30 seconds on condition that the electron beam output of 10kW, 40mV, 50mV of ionization current, 20A, and bias voltage-200V, and 0.5 micrometers of Ag film were formed.
[0038] When the load of 2 Ns, the radius of 6mm, and the ball-on disk trial of about 60000 rotations estimated abrasion resistance by making into the count of durable friction the count whose Ag film is lost, the count of durable friction was 250x102 times, and coefficient of friction was 0.45. No membranous isolation was seen in the same adhesion force evaluation as an example 1.
[0039] The conventional example 2 ... The 1st layer of an example 2 and this thickness was formed without using an electron beam type ion plating system for the same member as an example 2, and forming a hole, and the 2nd layer of Ag film was formed like the example 2. When it was similarly estimated as the example 2, the count of durable friction was 170x102 times, and coefficient of friction was 0.5. Moreover, about 30% of the film exfoliated in the same adhesion force evaluation as an example 1.
[0040] Example 4 ... 20 micrometers of CrN film were formed in the bearing and shaft of a high-speed pump like the example 1, and the front face was ground with the polish object with which the powder of SiC was distributed. Surface roughness Ra before polish was 0.20 micrometers, and after polish was 0.08 micrometers. 1 micrometer of MoS2 film was formed like the example 2 on it.
[0041] It finished setting up these bearings and a shaft on the pump, and they were rotated by 17m/s in axial rate by ordinary temperature underwater as an accelerated test. Consequently, most damages on membranous were not observed after the accelerated test of 24 hours.
[0042] Example of a comparison ... Except not forming MoS2 film, when it finished setting up the same bearing and same shaft as an example 4 on the pump and the same trial as an example 4 was carried out, membranous wear occurred in 30 minutes after a trial, backlash occurred, and the engine performance

was inadequate.
[0043]
[Effect of the Invention] by this invention, even if it uses cheap solid-state lubricating film, the adhesion force of solid-state lubricating film and a member does not decline, but low coefficient of friction obtains over a long time -- having -- in addition -- and the member with solid-state lubricating film which can hold high abrasion resistance was able to be offered.

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CLAIMS

[Claim(s)]
[Claim 1] The member with solid-state lubricating film in which the 1st layer of the hard film which has many detailed holes with a bore of 0.1-3 micrometers, and the 2nd layer of solid-state lubricating film were formed.
[Claim 2] The member with solid-state lubricating film according to claim 1 whose surface roughness Rmax of the 1st layer before forming the 2nd layer is 1 micrometer or less.
[Claim 3] The member with solid-state lubricating film according to claim 1 or 2 whose hard film of the 1st layer is the nitride film by the cathode arc ion plating method, the carbon nitride film, or carbide film.
[Claim 4] The member with solid-state lubricating film according to claim 1 to 3 whose solid-state lubricating film of the 2nd layer is MoS2 film formed by the ion plating method, the sputtering method, or the shot-peening method, WS2 film, NbS2 film, the mica film, 2OSb3 film, BN film, the WSe film, MoSe2 film, Au film, or Ag film.

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DERWENT-ACC-NO: 2000-479009
DERWENT-WEEK: 200042

TITLE: Material with solid lubrication layer formed with rigid film layer having fine holes with specific internal diameter and solid lubrication film layer

PATENT-ASSIGNEE: SUMITOMO METAL MINING CO (SUMM)

PRIORITY-DATA: 1998JP-0354090 (December 14, 1998)

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ABSTRACTED-PUB-NO: JP2000178720A
BASIC-ABSTRACT:

NOVELTY - A material with solid lubrication film comprises a layer of rigid film having fine holes with internal diameter of 0.1-3 microns and a solid lubrication film layer.

USE - As a machine part application e.g. abrasion component and tool.

ADVANTAGE - The adhesion strength of the solid lubrication film and material is maintained even if a cheap solid lubrication film is used but a low coefficient of friction is obtained over a long period of time. The material maintains a high antiwear quality.

ABSTRACTED-PUB-NO: JP2000178720A
EQUIVALENT-ABSTRACTS:

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(54)【発明の名称】　固体潤滑膜付き部材

(57)【要約】
【課題】　安価な固体潤滑膜を用いても固体潤滑膜と部材との密着力が低下せず、長時間にわたって低摩擦係数が得られ、しかも高い耐摩耗性が保持できる固体潤滑膜付き部材を提供する。
【解決手段】　内径０．１～３μｍの微細な孔を多数有する硬質膜の第１層と、固体潤滑膜の第２層が形成された固体潤滑膜付き部材。第２層形成前の第１層の表面粗さＲｍａｘを１μｍ以下とし、第２層の固体潤滑膜には、イオンプレーティング法、スパッタリング法、または、ショットピーニング法でMoS_2膜、WS_2膜、NbS_2膜、雲母膜、Sb_2O_3膜、ＢＮ膜、ＷＳｅ膜、$MoSe_2$膜、Ａｕ膜、または、Ａｇ膜などを形成する。

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【特許請求の範囲】

【請求項1】　内径0．1～3μmの微細な孔を多数有する硬質膜の第1層と、固体潤滑膜の第2層が形成された固体潤滑膜付き部材。

【請求項2】　第2層を形成する前の第1層の表面粗さRmaxが1μm以下である請求項1に記載の固体潤滑膜付き部材。

【請求項3】　第1層の硬質膜が、カソードアークイオンプレーティング法による窒化物膜、炭窒化物膜、または、炭化物膜である請求項1または請求項2に記載の固体潤滑膜付き部材。

【請求項4】　第2層の固体潤滑膜が、イオンプレーティング法、スパッタリング法、または、ショットピーニング法で形成されたMoS_2膜、WS_2膜、NbS_2膜、雲母膜、Sb_2O_3膜、BN膜、WSe膜、$MoSe_2$膜、Au膜、または、Ag膜である請求項1～請求項3のいずれかに記載の固体潤滑膜付き部材。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、潤滑性及び耐摩耗性に優れた固体潤滑膜付き部材に関し、より詳しくは、摺動摩耗部品や工具などの機械部品用途として有用な、低摩擦係数で耐摩耗性を向上した固体潤滑膜付き部材に関する。

【0002】

【従来の技術】工具や機械部品の摩擦係数を低減して耐摩耗特性を向上させる手段として、部材表面に固体潤滑膜を形成する方法が知られている。例えば、機械部品表面に油を塗布したり、潤滑微粒子を分散させたフィルムやオイルで覆ったり、スパッタリング法でMoS_2膜等どを形成したりする。

【0003】しかしこれら従来の方法では、固体潤滑膜が部材と拡散層を形成せず、部材と固体潤滑膜との密着力が弱かった。また、膜自体は耐摩耗性を有しないので、長時間使用していると膜が摩耗し、部材表面から剥離、摩耗して失われてしまうという欠点があった。

【0004】また、MoS_2などの固体潤滑膜は一般に密着力が弱くて吸湿性が高いので、膜自身が水分を吸着して粘性をもち、部材からすぐに剥離してしまいやすかった。

【0005】更に、高速回転するポンプの軸や軸受けなどでは特に高い耐摩耗性と高い潤滑性（低摩擦係数）が必要であり、さらに水中、熱水中で使用されるものは固体潤滑膜のより高い密着力を必要とする。このため上記従来の固体潤滑膜では特性が不十分であり、これまでは高価なセラミックス材や溶射材などを使用せざるを得なかった。

【0006】

【発明が解決しようとする課題】そこで本発明は、安価な固体潤滑膜を用いても固体潤滑膜と部材との密着力が

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低下せず、長時間にわたって低摩擦係数が得られ、しかも高い耐摩耗性が保持できる固体潤滑膜付き部材を提供することを目的とする。

【0007】

【課題を解決するための手段】上記目的を解決するための本発明の固体潤滑膜付き部材は、内径0．1～3μmの微細な孔を多数有する硬質膜の第1層と、固体潤滑膜の第2層が形成されたことを特徴とする。

【0008】本発明の固体潤滑膜付き部材では、第2層を形成する前の第1層の表面粗さRmaxが1μm以下であることが望ましい。

【0009】第1層の硬質膜は、カソードアークイオンプレーティング法による窒化物膜、炭窒化物膜、または、炭化物膜であることが好ましい。カソードアークイオンプレーティング法では、バイアス電圧を－500～－1500Vとするとよい。

【0010】第2層の固体潤滑膜は、イオンプレーテイング法、スパッタリング法、または、ショットピーニング法で形成されたMoS_2膜、WS_2膜、NbS_2膜、雲母膜、Sb_2O_3膜、BN膜、WSe膜、$MoSe_2$膜、Au膜、または、Ag膜であることが好ましい。イオンプレーテイング法、スパッタリング法、または、ショットピーニング法では、バイアス電圧を－500～－1500Vとするとよい。

【0011】

【発明の実施の形態】本発明の固体潤滑膜付き部材の部材には、従来から摺動摩耗部品や工具などの機械部品用途に従来から用いられているものが適用できる。例えば、切削工具、金型用のSKD鋼、SKH鋼、SKS鋼、機械構造用のSC鋼などである。

【0012】部材表面には、第1層として内径0．1～3μmの微細な孔を多数有する硬質膜を形成するが、これは硬質膜と固体潤滑膜との密着性を向上させることを目的として形成するものである。

【0013】硬質膜表面に0．1～3μmの微細な孔（凹み）を多数形成すれば、その凹みを核として固体潤滑膜が付着し、アンカー効果によって密着力が向上するのである。また、固体潤滑膜が摩耗されても、凹みに堆積している固体潤滑膜が相手材と接触し、掘り起こされ、染み出すことにより、部材表面の固体潤滑性が長時間保たれることにもなる。

【0014】微細な孔の内径を0．1～3μmとするのは、この範囲の内径とすることで固体潤滑膜が孔に入り込み、表面から容易に除去されないからである。また、この微細な孔は、膜厚が5μmで面積当たり0．01%、10～20μmで0．001%程度の存在量が好ましい。

【0015】硬質膜は、望ましくは窒化物、炭窒化物、または、炭化物からなるもので、具体的にはCrN、TiN、TiCN、TiAlNなどを用いることができ、

このような材質とすることで、耐摩耗性と低摩擦係数が得られる。硬質膜は、例えばカソードアーク式イオンプレーティング法で形成することができる。

【0016】このカソードアーク式イオンプレーティング法は、蒸発源である金属ターゲットを陰極とし、チャンバーを陽極として、これらの間にアーク放電を起こして金属蒸気をイオン化させ、一方で反応ガスをイオンとの衝突によりイオン化し、金属イオンと反応ガスイオンとで生成した化合物を部材表面に積層させて膜を形成するものである。例えば窒化膜を形成する場合は、チャンバー内に窒素原子を含む反応ガスを導入すればよい。

【0017】カソードアーク式イオンプレーティング法は、金属のイオン化率が高く、複数の蒸発源をチャンバー内の上下左右に設置できるので、複合膜や、大型形状や複雑形状の部材に均一な厚さの膜が形成でき、これら部材の硬質膜形成に向いている。膜形成前に、ターゲットと部材間にかけるバイアス電圧を－５００～－１５００Vとすると、金属が部材に付着し、最大粗さ５μmの凹凸となる。この上に窒化膜等の硬質膜を成膜すれば、内径０．１～３μmの多数の孔が形成できる。

【0018】バイアス電圧を－５００V未満と低くすると、孔の数が減少し、本発明の効果が少なくなる。一方、バイアス電圧を－１５００Vを超えて高くすると、孔の数は増加するが、部材の表面粗さも増加し、製品としての寸法精度が落ちてくる。

【0019】第２層には固体潤滑膜を第１層の上に形成する。固体潤滑膜の材料には、硫化物のMoS_2、WS_2、NbS_2、酸化物の雲母、Sb_2O_3、窒化物のBN、セレン化物のWSe、$MoSe_2$、金属のAu、Agなどが挙げられる。この固体潤滑膜は、例えばイオンプレーティング法、スパッタリング法、ショットピーニング法などで形成すればよい。

【0020】第２層を形成する前の第１層の表面粗さRmaxは、表面研磨処理等により、望ましくは１μm以下とする。これにより、「ドロップレット」といわれる硬質膜表面に形成される溶融金属を除去、減少でき、硬質膜と固体潤滑膜との接触面積が増えて密着力が向上するからである。また、ドロップレットが膜から剥離して固体潤滑膜を削ってしまうことも防げる。表面研磨処理としては、ダイヤモンドペーパー研磨、平面研削機、バフ研磨等が利用できる。

【0021】固体潤滑膜は、硬質膜に形成された孔の内部に入り込み、部材と固体潤滑膜の密着力をアンカー効果により機械的に結合する。この結合力は、従来方法より強くなる。

【0022】スパッタリング法で固体潤滑膜を形成場合は更に、直流（DC）スパッタリングや、高周波（RF）スパッタリング法を用いることができる。放電ガスにはNe、Ar、Kr等の不活性ガスが利用できるが、取り扱いが容易であるArガスが好ましい。例えば放電ガス圧を１～４Paに設定すれば、緻密で密着力が高い固体潤滑膜が形成される。ターゲットに対する放電電力は、ターゲット当たり１００～５００Wとすればよい。

【0023】固体潤滑膜の膜厚は、０．１～４μmとすることが望ましい。０．１μm未満では十分な潤滑性は得られず、４μmを超えると膜のせん断が起こりやすくなり、膜中に多くのAr分子を含有するので潤滑特性も劣化してくるからである。

【0024】ショットピーニング法で固体潤滑膜を形成する場合は、固体潤滑剤粉末と合成樹脂粒子とを一定割合で混合した混合体を、乾式ブラスト装置により部材表面に噴射すればよい。固体潤滑剤粉末は部材表面に衝突して付着し、樹脂粒子は表面に衝突すると同時に反発して気体流とともに飛び去る。このとき樹脂粒子は、表面に付着した固体潤滑粉体を打ち込みかつ擦り込み、表面との密着性の良い固体潤滑膜が形成される。なお、ショットピーニング法においては固体潤滑膜の厚膜を形成しにくいので、厚さ１μm以下が適当である。

【0025】

【実施例】実施例１　・・・　厚さ２mm、２０mm角の工具鋼（SKH51）を部材とし、マルチアーク社製カソードアーク式イオンプレーティング装置を用いて第１層の膜を形成した。ターゲットにはCr金属を用いた。部材をエタノール中で超音波洗浄した後、真空チャンバー内にセットし、チャンバー内を2×10^{-5}Torr以下まで排気した。ターゲットと部材間に－１０００Vのバイアス電圧を印加し、Cr金属のメタルボンバードで部材表面を洗浄し、加熱してCrイオンの打ち込みを行い、部材温度を４５０℃にした。

【0026】次に、チャンバー内にN_2ガスを５０mTorrまで導入し、バイアス電圧－３００VでCrN膜を１０μm形成し第１層とした。

【0027】形成したCrN膜の表面を観察すると、１mm^2当たり、内径０．２～３μmの孔が１００～２００個形成されていた。

【0028】続いて、RFマグネトロンスパッタリング装置（日電アネルバ株式会社製、SPF530H）を用いて、第２層の潤滑剤膜を形成した。装置内の所定位置に、直径５インチの円盤型二硫化モリブデン（MoS_2）ターゲットをセットし、チャンバー内の真空度が5×10^{-4} Paになるまで真空引きし、Arガスを０．４Paまで導入した。放電電力は３００W（２．３７W／cm^2）投入し、MoS_2をArガス雰囲気で１．２μm形成して第２層とした。

【0029】得られた固体潤滑膜付き部材を、直径６mmのSUS440C製のボールを用い、荷重１０N、周速度０．５m／secとした直径１２mmのボールオンディスク試験で、MoS_2が無くなる回数を耐久摩擦回数として評価した。耐久摩擦回数は450×10^2回で、摩擦係数は０．１であった。また、ビッカース硬度

は１８００HVであった。また、密着力を、部材表面にカッターナイフを用いて間隔１mm、長さ約１０mmの傷を格子状に形成し、その部分に粘着テープを貼付し、これを一気に剥がして膜の剥離を観察したところ、剥離は一切見られなかった。

【００３０】従来例１　・・・　実施例１と同じ部材に、電子ビーム式イオンプレーティング装置を用いて孔を形成せずに実施例１と同厚みの第１層を形成し、実施例１と同様にしてMoS_2の第２層を形成した。実施例１と同様に評価したところ、耐久摩擦回数は２７０×10^2回で、摩擦係数は０．１５であった。また、ビッカース硬度は９００HVであった。実施例１と同様の密着力評価では、膜の約５０%が剥離した。

【００３１】実施例２　・・・　厚さ２mm、２０mm角のステンレス鋼（ＳＵＳ３０４）を部材とし、ＣｒＮ膜を２０μmとした以外は実施例１と同様にして第１層の膜を形成した。形成したＣｒＮ膜の表面を観察すると、１mm^2当たり、内径０．２～２μmの孔が１００～２００個形成されていた。

【００３２】この部材表面をバフ研磨して、表面粗さＲｍａｘを０．０８μmにした。この研磨した部材に、MoS_2粉末７４.０gと５０μmの樹脂ビーズ３２０gとの混合粉を、噴射エアーガンと部材との距離を１００mm、噴射エアー圧を３kgf/cm^2にして吹き付け、ショットピーニング法によりMoS_2膜からなる第２層膜を形成した。第２層の膜厚は１μmであった。

【００３３】実施例１と同様の密着力評価では、膜の隔離は一切見られなかった。

【００３４】また、荷重２００kgf、回転速度５０rpmのファレックス試験では、回転開始から１０００秒経過してもトルク値はほとんどかわらず、固体潤滑膜も剥離しなかった。

【００３５】従来例１の部材を同様にファレックス試験で評価したところ、回転開始後MoS_2粉末が飛び散り、８００秒経過後からトルク値が徐々に増加した。

【００３６】実施例３　・・・　実施例１と同様に第１層としてＣｒＮ膜を１０μm形成し、表面に発生しているドロップレットをバフ研磨で取り去って評価した。バフ研磨前の表面粗さＲａは０．０８μm、バフ研磨後は０．０３μmであった。

【００３７】その上に、神港精機製ピアス式イオンプレーティング装置により、Ａg膜からなる固体潤滑膜を形成した。銅－モリブデン製ルツボに台形型のＡgインゴットを入れ、電子ビーム出力１０kW、４０mV、イオン化電流５０mV、２０A、バイアス電圧－２００Vの条件で３０秒成膜し、Ａg膜を０．５μm形成した。

【００３８】荷重２N、半径６mm、約６００００回転のボールオンデイスク試験により、Ａg膜が無くなる回数を耐久摩擦回数として耐摩耗性を評価したところ、耐久摩擦回数は２５０×10^2回、摩擦係数は０．４５であった。実施例１と同様の密着力評価では、膜の隔離は一切見られなかった。

【００３９】従来例２　・・・　実施例２と同じ部材に、電子ビーム式イオンプレーティング装置を用いて孔を形成せずに実施例２と同厚みの第１層を形成し、実施例２と同様にしてＡg膜の第２層を形成した。実施例２と同様に評価したところ、耐久摩擦回数は１７０×10^2回で、摩擦係数は０．５であった。また、実施例１と同様の密着力評価では、膜の約３０%が剥離した。

【００４０】実施例４　・・・　高速ポンプの軸受け及び軸に、実施例１と同様にしてＣｒＮ膜を２０μm形成し、表面をＳｉＣの粉が分散された研磨体で研磨した。研磨前の表面粗さＲａは０．２０μm、研磨後は０．０８μmであった。その上に実施例２と同様にしてMoS_2膜を１μm形成した。

【００４１】これら軸受け及び軸をポンプに組み上げ、加速試験として、常温水中で軸速度１７m／ｓで回転させた。その結果、２４時間の加速試験後でも膜の損傷はほとんど観察されなかった。

【００４２】比較例　・・・　MoS_2膜を形成しない以外は実施例４と同様の軸受け及び軸をポンプに組み上げ、実施例４と同様の試験をしたところ、試験後３０分で膜の摩耗が発生してガタが起き、性能が不十分だった。

【００４３】

【発明の効果】本発明により、安価な固体潤滑膜を用いても固体潤滑膜と部材との密着力が低下せず、長時間にわたって低摩擦係数が得られ、なおかつ高い耐摩耗性が保持できる固体潤滑膜付き部材を提供することができた。

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